

# CHESTER LabNet

EFFECTIVE DATE: 7-3-03

---

Chester LabNet

**Standard Operating Procedure  
XR-004.01**

**KEVEX XRF SPECTROMETER CALIBRATION  
CHESTER LABNET PROPRIETARY METHOD**

---

Approvals:

  
AUTHOR

7-1-03  
DATE

  
TECHNICAL REVIEWER

7-3-03  
DATE

  
QUALITY ASSURANCE

7/2/03  
DATE

  
LABORATORY SUPERVISOR

7-3-03  
DATE

## REVIEW HISTORY

<u>Review date:</u>	<u>Changes made:</u>	<u>Changes made by:</u>
7/1/03	Minor changes to text.	Sheri Heldstab
4/22/02	No changes. Date of origination. Original text found in SOP XR-002, written by Rick Sarver in 1998.	Sheri Heldstab

## ANNUAL REVIEW

The undersigned attests that this standard operating procedure has undergone annual review for adherence to current practices and the latest QA/QC protocols:

\_\_\_\_\_  
signature

\_\_\_\_\_  
printed name/title

\_\_\_\_\_  
date

**KEVEX-770 SPECTROMETER CALIBRATION  
CHESTER LABNET PROPRIETARY METHOD**

**1.0 Scope and Application**

- 1.1 This method is applicable to the calibration of Kevex Model Delta 770 energy dispersive XRF (EDXRF). Chester LabNet currently operates two of these instruments, informally named “770” and “772”. The process by which each instrument is calibrated is identical.

**2.0 Summary**

- 2.1 The instruments are configured for the analytes of interest, then a series of standards and matrices are analyzed to determine the peak energies of each analyte. A response factor for each element at each excitation utilized condition is determined, and that factor is entered into the IXRF operational software.
- 2.2 Also determined are interelemental interferences and matrix differences. These factors are entered into the LabNet proprietary software, which corrects for these issues.
- 2.3 Full calibration will take a minimum of 2 working days to complete.
- 2.4 Calibration should be performed only by trained operators with experience in data interpretation and instrument functioning. This SOP is designed only as a guide, and should not be used in lieu of proper training.

**3.0 Sampling and Storage**

- 3.1 Standards may be stored at room temperature, as analysis is performed at room temperature, and under vacuum.

**4.0 Apparatus**

- 4.1 Instrument. Kevex Model Delta 770 energy dispersive XRF (EDXRF) equipped with a water-cooled end-window X-ray tube with a rhodium anode and a peak operating power of 60kV and

3.3 mA. The system was modified by IXRF Inc. to operate using Windows based software to control instrument operation. Chester LabNet currently operates two of these instruments, informally named "770" and "772". 770 is the original instrument. Its computer operates on Windows 3.1 and uses floppy discs to transfer data to the data processing computer. 772 operates on Windows 98 and uses an Ethernet connection to transfer data files.

#### 4.2 Thin Film Calibration Standards

- 4.2.1 Micromatter Inc. vapor deposit of single or two non-interfering elements onto thin mylar film.
- 4.2.2 EPA organo-metallic acetate film, usually with two non-interfering elements.
- 4.2.3 NIST SRM's 1832 and 1833 multi-element vapor deposits on glass films.

### 5.0 Reagents

5.1 95% ethanol

5.2 Liquid Nitrogen

### 6.0 Calibration/Preparation

6.1 Ensure the proper excitation conditions are configured within the IXRF software. Different excitation conditions are used in order to maximize the sensitivity of the analysis over a wide range of elements fluorescing at various energies. If only one energy is listed below, it is used for both instruments. The analytes appearing in **bold** type are in their most favorable excitation condition.

6.1.1 Excitation conditions for 'low-density' filter media (Teflon, Nuclepore, Mylar).

6.1.1.1 sp0: direct Rh  $L\alpha$  excitation with one thickness of Whatman no. 41 cellulose used as a prefilter to absorb low-energy X-rays from source. Energy: 7.5 kV. Analytes of interest: **Na, Mg, Al, Si, P, S**. Tube geometry (incident beam angle):  $\theta = 21^\circ$ .

- 6.1.1.2 sp1: Ti secondary target. Energy: 25 kV. Analytes of interest: Al, Si, P, **S, Cl, K, Ca**. Tube geometry:  $\theta = 45^\circ$
- 6.1.1.3 sp2: Fe secondary target. Energy: 35 kV for 770, 25kV for 772. Analytes of interest: K, Ca, **Ti, V, Cr**. Tube geometry:  $\theta = 45^\circ$
- 6.1.1.4 sp3: Ge secondary target. Energy 35 kV for 770, 30kV for 772. Analytes of interest: K, Ca, Ti, V, Cr, **Mn, Fe, Co, Ni, Cu, Zn, Sm, Eu, Tb**. Tube geometry:  $\theta = 45^\circ$
- 6.1.1.5 sp4: direct Rh  $K\alpha$  excitation with .1 mm Rh foil prefilter. Energy: 35 kV. Analytes of interest: K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, **Ga, Ge, As, Se, Br, Rb, Sr, Y, Zr, Nb, Mo, Hf, Ta, W, Ir, Au, Hg, Pb**. Tube geometry:  $\theta = 21^\circ$
- 6.1.1.6 sp5: direct Rh bremsstrahlung excitation with .1 mm W foil prefilter. Energy: 55 kV. Analytes of interest: **Rh, Pd, Ag, Cd, In, Sn, Sb, Te, I, Cs, Ba, La, Ce**. Tube geometry:  $\theta = 21^\circ$
- 6.1.2 Excitation conditions for 'high-density' filter media (quartz, glass fiber, carbon impregnated).
- 6.1.2.1 sp0: Fe secondary target. Energy: 35 kV for 770, 25kV for 772. Analytes of interest: **Al, Si, P, S, Cl, K, Ca, Ti, V, Cr**. Tube geometry:  $\theta = 45^\circ$
- 6.1.2.2 sp1: Ge secondary target. Energy 35 kV for 770, 30kV for 772. Analytes of interest: K, Ca, Ti, V, Cr, **Mn, Fe, Co, Ni, Cu, Zn**. Tube geometry:  $\theta = 45^\circ$
- 6.1.2.3 sp2: direct Rh  $K\alpha$  excitation with .1 mm Rh foil prefilter. Energy: 35 kV. Analytes of interest: K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, **Ga, Ge, As, Se, Br, Rb, Sr, Y, Zr, Mo, Hg, Pb**. Tube geometry:  $\theta = 21^\circ$
- 6.1.2.4 sp3: direct Rh bremsstrahlung excitation with .1 mm W foil prefilter. Energy: 55 kV. Analytes of interest: **Rh, Pd, Ag, Cd, In, Sn, Sb, Te, I,**

**Cs, Ba, La, Ce.** Tube geometry:  $\theta = 21^\circ$

6.2 Preparation of standards for analysis: standards are permanently mounted in sample holders and stored in a tray labeled "standards" in the XRF lab.

6.3 Characterizing peak energies:

6.3.1 Peak energies should only have to be determined if the instrument is brand new or if the X-ray power supply of the instrument has been replaced. Characterization of peak energies should ONLY be performed by analysts with extensive knowledge of the instrument. The description below is only a brief overview of the technique.

6.3.2 Operate the XRF in manual mode.

6.3.3 Set the system up as follows:

6.3.3.1 Pressure = vacuum

6.3.3.2 Energy = defined by elements of interest

6.3.3.3 mA and kV set to approximate that needed by given element

6.3.3.4 set the filter appropriately for the element of interest

6.3.3.5 set the target appropriately for the element of interest.

6.3.3.6 Start the X-rays and allow to run until the peak is of the desired height.

6.4 Obtaining Calibration Counts:

6.4.1 Analyze each standard following the protocols in SOP XR-002 (ELEMENTAL ANALYSIS OF AIR FILTERS BY X-RAY FLUORESCENCE USING A KEVEX-770 SPECTROMETER AND IXRF OPERATING SYSTEM)

6.4.2 Use the QS analytical protocol to analyze the standards at reduced power (mA).

6.4.3 After transferring the raw data into the excel program, determine the net counts.

## 7.0 Procedure

7.1 Determine energies for each analyte/excitation condition. Energy calibration is performed using Kevex Standard No. 2036 and the Kevex software calibration option in which a low energy line is measured at the Al K $\alpha$  line (1.487 KeV) and a high energy line is measured at the Cu K $\alpha$  line (8.047 KeV). The program will then assign channel number 149 as the Al peak centroid, and channel 805 as the Cu peak centroid, thereby calibrating each channel increment at 10 eV.

7.2 Identify KeV location of peaks: Peak calibration is performed by analyzing thin film standards containing the elements of interest (see section 4.2). Peaks should be gaussian-shaped with at least 2000 counts in the central channel (centroid).

7.2.1 Determine channel numbers associated with each analyte:

7.2.1.1 Calculate the Full-Width-at Half-Maximum (FWHM) window for each element by dividing the number of counts in the most intense channel (usually the centroid) by two (HM).

7.2.1.2 Next, find the channel on the low energy side of the peak closest in counts to HM = LEHM (low-energy-at half-max).

7.2.1.3 Repeat this procedure for the HEHM (high-energy-at half-max).

7.2.1.4 The area between and including LEHM and HEHM represents the FWHM window for that element where LEHM and HEHM are defined as channel numbers. The sum of the counts in these channels should represent approximately 70% of the total area of the peak.

7.2.2 Determine peak centroids associated with analytes of interest:

7.2.2.1 Determine the peak centroid for each analyte contained in the standard by the following equation:  $(LEHM + HEHM) / 2$ .

7.2.2.2 Plot a graph of atomic number along the x-axis, versus peak centroid on the y-axis.

7.2.2.3 This graph should yield a smoothly varying curve sloping upward.

7.2.2.4 Visual interpretation of the graph, or algebraic regression can be used to approximate peak positions for elements not represented by standards.

7.2.3 Determine peak widths associated with analytes of interest:

7.2.3.1 Determine the peak width for each analyte contained in the standard by the following equation:  $(\text{HEHM} - \text{LEHM}) + 1$ .

7.2.3.2 Plot a graph of atomic number along the x-axis, versus peak width on the y-axis.

7.2.3.3 This graph should yield a smoothly varying curve sloping upward.

7.2.3.4 Visual interpretation of the graph, or algebraic regression can be used to approximate peak widths for elements not represented by standards.

7.2.3.5 Note that separate plots should be made for  $K\alpha$ ,  $L\alpha$ , and  $L\beta$  peaks.

7.2.3.6 Some peak widths and centroids for LabNet's 770 Kevex instrument are:

Analyte line	centroid	width
	(channel #)	(no. of channels)
Si $K\alpha$	174.5	12
Fe $K\alpha$	640.5	16
Pb $L\beta$	1263.0	19
Cd $K\alpha$	2312.5	32
La $K\alpha$	3334.0	53

7.3 Determine calibration response factors for each analyte of interest:

7.3.1 Determine the calibration factor for each analyte normalized to 1.0 mA. See Section 9.4 for calculations.



7.3.2 Plot the calibration curves associated with analytes of interest:

7.3.2.1 Determine the sensitivity factor at 1mA for each analyte contained in the standard by the following equation:  $S_n = (1 / \text{Calibration Factor})$ .

7.3.2.2 Plot a graph of atomic number along the x-axis, versus the log of the sensitivity factor on the y-axis.

7.3.2.3 This graph should yield a smoothly varying curve sloping upward, except for Tungsten filtered 55 kV, which slopes downward.

7.3.2.4 Visual interpretation of the graph, or algebraic regression can be used to approximate calibration factors for elements not represented by standards as long as the atomic number of the analyte is within the range represented on the graph. Sensitivity values lying outside the curve should be checked by analyzing additional standards

7.3.2.5 Note that separate plots should be made for  $K\alpha$ ,  $L\alpha$ , and  $L\beta$  peaks.

7.4 Calculate interference factors from the thin film standards used for calibration using the equations in section 9.6.

7.5 Determine the correction for  $PM_{2.5}$  particle size for Aluminum.

7.5.1 Analyze the 'fine' fractions of particulate matter deposited onto 37mm Teflon filters in the form of  $Al_2O_3$  and  $Al_2Si_2O_7$ .

7.5.2 Create the deposits by suspending the particulate matter and 'sampling' with a Sierra-Andersen dichotomous sampler. See SOP XR-001 for suspension of particulate matter onto filters.

7.5.3 The particle size attenuation effect for Al is then calculated for each standard following the equations in section 9.7.

7.6 Update the Chester LabNet XRF Data Processing Program with the newly acquired factors as follows:

7.6.1 The peak location data are entered into the file labeled 'peaks' within the LabNet XRF Data Processing Program.

7.6.1.1 The first column contains the element of interest.

7.6.1.2 The next four columns define:

7.6.1.2.1 the low-energy-background (LEB), where L = the low-energy channel,

7.6.1.2.2 H = the high energy channel,

7.6.1.2.3 C = the LEB centroid; and

7.6.1.2.4 # denotes the number of channels in the LEB window.

7.6.1.3 The next four columns represent the analyte peak and the following four columns labeled 'HEB' define the high-energy-background channels.

7.6.2 The calibration factors data are entered into the file labeled 'peaks' within the LabNet XRF Data Processing Program.

7.6.2.1 The column at the far right contains the calibration factors for the elements of interest normalized to 1.0 mA current.

7.6.2.2 The next column to the left contains the calibration factors normalized to the current (mA) used in the analysis of that excitation condition.

7.6.3 Enter the interference factors into the file labeled 'IEC' within the LabNet XRF Data Processing Program.

7.6.4 Enter the size correction factor for Al into the size/absorption correction algorithm in the LabNet XRF Data Processing Program.

## 8.0 QA/QC

8.1 Curve shapes for all graphs: All graphs of analyte atomic number vs other parameter should yield a smooth quadratic line. If the graph is not quadratic, all operating parameters must be verified and the calibration performed from the beginning.

## 9.0 Calculations

9.1 Full-Width-at Half-Maximum (FWHM) window. The area between and including LEHM and HEHM represents the FWHM window for that element.

HM = [counts in the most intense channel (usually the centroid)]  $\div$  2.

LEHM = [channel on the low energy side of the peak closest in counts to HM]

HEHM = [channel on the high energy side of the peak closest in counts to HM]

where: HM = counts at half maximum

LEHM = Low-energy-half-maximum channel number

HEHM = High-energy-half-maximum channel number

Note that the sum of the counts in these channels should represent approximately 70% of the total area of the peak.

9.2 Peak centroid channel number:

$$\text{Centroid} = (\text{LEHM} + \text{HEHM}) / 2$$

where: LEHM = Low-energy-half-maximum channel number

HEHM = High-energy-half-maximum channel number

9.3 The peak width in channels:

$$\text{Width} = (\text{HEHM} - \text{LEHM}) + 1$$

where: LEHM = Low-energy-half-maximum channel number

HEHM = High-energy-half-maximum channel number

9.4 Calibration factors are calculated as follows:

$$I_n = I_g - I_b$$

where:  $I_n$  = net counts

$I_g$  = the gross counts (sum of all counts in FWHM window)

$I_b$  = the background counts (average bkg cts x peak width)

$$F = V / (I_n / t)$$

where:  $F$  = the calibration factor  $[(\mu\text{g}/\text{cm}^2)/(\text{counts}/\text{second})]$

$t$  = the counting livetime (seconds)

$V$  = the surface equivalent value (SEV) of the element in the standard.

SEV is the absorption corrected value. Absorption effects will be discussed later in the text.

The calibration factor is then normalized to 1.0 mA as follows:

$$F_n = F / C_c$$

where:  $F_n$  = the calibration factor at 1.0 mA

$C_c$  = the current (mA) used during calibration

9.5 Sensitivity factors are calculated as follows:

$$S_n = 1 / F_n \text{ where } S_n \text{ represents the sensitivity factor at 1.0 mA}$$

9.6 Interference factors are calculated for spectral overlap using the data from the thin film standards used for calibration:

$$L_i = N_i / N_j$$

where:  $L_i$  = the interference correction factor (cts/s in the window of element i due to element j)

$N_i$  = net cts/s in window of element i

$N_j$  = net cts/s in window of element j (interfering element)

Most spectral overlap is the result of the  $K\beta$  line of element j overlapping the  $K\alpha$  line of element i. Another common interference occurs when an L-line of element j from one excitation condition overlaps the  $K\alpha$  line of element i in a separate excitation condition. In the latter case it is necessary to normalize the correction factor (L) to 1.0 mA per second as follows:

$$L_i = (N_i / C_i T_i) / (N_j / C_j T_j)$$

where:  $C_i$  = the current (mA) used in the excitation condition in which element i is measured.

$C_j$  = the current (mA) used in the excitation condition in which element j is measured.

$T_i$  = livetime (sec) for the excitation condition in which the element is measured.

$T_j$  = livetime (sec) for the excitation condition in which element j is measured.

9.7 The mass absorption effect for Al is then calculated for each particle size attenuation standard as follows:

#### 9.7.1 Mass absorption coefficient

$$\mu = \sum (\mu_i w_i)$$

where:  $\mu$  = the total mass absorption coefficient of all elements present on the incoming X-rays ( $\text{cm}^2/\text{g}$ )

$\mu_i$  = the mass absorption coefficient of each element i on the incoming X-rays

$w_i$  = the weight percent of element i in the deposit ( $0 < w_i < 1$ )

#### 9.7.2 Total Mass absorption coefficient

$$\mu' = \sum (\mu_{i,j} w_i)$$

where:  $\mu'$  = the total mass absorption coefficient of all elements present on the exiting (fluorescing) X-rays from element j

$\mu_{ij}$  = the mass absorption coefficient of each element i on the exiting X-rays from element j.

### 9.7.3 Total Mass absorption coefficient by area

$$\underline{\mu} = (\mu \csc \theta) + (\mu' \csc \theta')$$

where:  $\underline{\mu}$  = the total mass absorption coefficient ( $\text{cm}^2/\text{g}$ )

$\theta$  = the angle at which the incoming X-rays strike the sample

$\theta$  is described in sections 6.1 and 6.2 for each excitation condition

$\theta'$  = the angle at which the exiting X-rays travel to the detector and is fixed at  $45^\circ$

### 9.7.4 Mass absorption:

$$A = (1 - e^{-\underline{\mu}m}) / \underline{\mu}m$$

where: m = the areal density of the deposit ( $\text{g}/\text{cm}^2$ )

A = mass absorption

e = natural log exponent (mathematical e)

### 9.7.5 The net counts are then corrected for the calculated absorption for each standard as follows:

$$N_c = N / A$$

where:  $N_c$  = the absorption corrected intensity (net counts/second)

N = the uncorrected net count counts/second

### 9.7.6 Determine final size correction:

9.7.6.1 Plot the empirical concentration of Al for each standard along the x-axis versus the absorption corrected intensity along the y-axis.

9.7.6.2 Perform a linear regression, forcing the line through zero, such that

$$y = mx.$$

where:  $m$  = the sensitivity factor  $[(\text{cts/s})/(\mu\text{g}/\text{cm}^2)]$ .

9.7.6.3 Calculate the size correction factor as follows:

$$A_{i,p} = S_t / S_p$$

where:  $A_{i,p}$  = the size attenuation factor for element  $i$  at particle size  $p$

$S_t$  = the thin film calibration sensitivity factor for element  $i$

$S_p$  = the particle standard sensitivity factor which is the slope of the line derived from the linear regression above.

Simply stated, the attenuation for mass absorption has been taken into account, so that if there were no additional attenuation due to the size of the particles, then  $S_p = S_t$  and  $A_{i,p} = 1$ . Any additional attenuation would then be due to individual particle size, and  $A_{i,p} < 1$ .  $A_p \cong 0.667$  for Al  $\text{PM}_{2.5}$ , and  $A_p \cong 1$  for Si  $\text{PM}_{2.5}$ .

## 10.0 References

- 10.1 Kevex Operator's Manual
- 10.2 Practical X-Ray Spectrometry, R. Jenkins and J.L. De Vries, second edition, Philips Technical Library, Springer-Verlag New York Inc.
- 10.3 X-Ray Fluorescence Analysis of Environmental Samples, Jaklevic, et al, Ed. by T.G. Dzubay, Ann Arbor Sci.
- 10.4 Self Absorption Corrections for X-Ray Fluorescence Analysis of Aerosols, T.G. Dzubay and R.O. Nelson, in Advances in X-Ray Analysis, Vol 18, 619-631.
- 10.5 Quantitative Analysis of Aluminum and Silicon in Air Particulate Deposits on Teflon Membrane Filters by X-Ray Fluorescence Analysis, J.A. Cooper, L.M. Valdovinos, J.R. Sherman, W.L. Pollard, R.H. Sarver, and J.K. Weider, report by NEA, Inc., Beaverton, OR,

July 15, 1987.

10.6 Round Robin Evaluation: Elemental Analysis of Bulk Samples and PM<sub>10</sub> Loaded Teflon Filters, presented at the 82nd Annual Meeting of AWMA, Anaheim, CA, June 24-30, 1989.

**11.0 Analyst Notes**

11.1 N/A



SP0	LEB				Analyte				HEB				Avg Counts				Calibration	
	L	H	C	#	L	H	C	#	L	H	C	#	LEB	HEB	m	b	Factors	
Na	91	93	92	3	95	110	102.5	16	114	116	115	3	69.3	61.0	-0.3623	102.6667	0.04273	0.03205
Mg	114	116	115	3	118	133	125.5	16	133	134	133.5	2	61.0	67.0	0.3243	23.7027	0.01930	0.01447
Al	137	139	138	3	142	157	149.5	16	158	160	159	3	47.7	60.0	0.5873	-33.3810	0.00932	0.00699
Si	158	160	159	3	167	182	174.5	16	188	191	189.5	4	60.0	79.5	0.6393	-41.6557	0.00516	0.00387
P	191	192	191.5	2	193	209	201.0	17	210	211	210.5	2	78.0	98.5	1.0789	-128.6184	0.00336	0.00252
S	199	201	200	3	223	239	231.0	17	249	251	250	3	75.7	179.3	2.0733	-339.0000	0.00245	0.00184
<b>SP1</b>																		
Al	113	117	115	5	142	157	149.5	16	298	302	300	5	88.8	63.6	-0.1362	104.4649	0.19094	0.57283
Si	113	117	115	5	167	182	174.5	16	298	302	300	5	88.8	63.6	-0.1362	104.4649	0.10518	0.31555
P	113	117	115	5	193	209	201.0	17	298	302	300	5	88.8	63.6	-0.1362	104.4649	0.06828	0.20484
S	113	117	115	5	223	239	231.0	17	298	302	300	5	88.8	63.6	-0.1362	104.4649	0.04921	0.14764
Cl	113	117	115	5	254	270	262.0	17	298	302	300	5	88.8	63.6	-0.1362	104.4649	0.03526	0.10577
K	298	302	300	5	324	341	332.5	18	414	416	415	3	63.6	208.3	1.2586	-313.9652	0.01943	0.05829
Ca	298	302	300	5	361	378	369.5	18	414	416	415	3	63.6	208.3	1.2586	-313.9652	0.01460	0.04381
Ti MS	113	117	115	5	443	460	451.5	18	524	528	526	5	88.8	11.4	-0.1883	110.4569		
<b>SP2</b>																		
K	298	302	300	5	324	341	332.5	18	421	425	423	5	78.2	55.4	-0.1854	133.8098	0.01706	0.05117
Ca	298	302	300	5	361	378	369.5	18	421	425	423	5	83.2	55.4	-0.2260	151.0049	0.01300	0.03900
Sc	298	302	300	5	401	418	409.5	18	421	425	423	5	83.2	55.4	-0.2260	151.0049	0.01026	0.03077
Ti	421	425	423	5	443	460	451.5	18	517	521	519	5	55.4	85.4	0.3125	-76.7875	0.00809	0.02427
V	421	425	423	5	487	505	496.0	19	517	521	519	5	55.4	85.4	0.3125	-76.7875	0.00633	0.01900
Cr	512	521	516.5	10	533	551	542.0	19	564	573	568.5	10	79.4	123.7	0.8519	-360.6183	0.00491	0.01474
Fe MS	316	320	318	5	631	650	640.5	20	737	741	739	5	77.8	24.6	-0.1264	117.9843		
<b>SP3</b>																		
Cr	420	424	422	5	533	551	542.0	19	564	568	566	5	41.4	21.4	-0.1389	100.0111	0.01279	0.03838
Mn	564	568	566	5	581	599	590.0	19	726	730	728	5	21.4	27.6	0.0383	-0.2617	0.01033	0.03100
Fe	564	568	566	5	631	650	640.5	20	726	730	728	5	21.4	27.6	0.0383	-0.2617	0.00826	0.02478
Co	564	568	566	5	684	703	693.5	20	726	730	728	5	21.4	27.6	0.0383	-0.2617	0.00660	0.01981
Ni	667	671	669	5	738	758	748.0	21	767	771	769	5	20.8	27.0	0.0620	-20.6780	0.00563	0.01690
Cu	767	771	769	5	795	815	805.0	21	833	837	835	5	27.0	44.0	0.2576	-171.0758	0.00483	0.01450
Zn	833	837	835	5	854	875	864.5	22	883	887	885	5	44.0	102.8	1.1760	-937.9600	0.00404	0.01211
Sm	516	520	518	5	554	572	563.0	19	726	730	728	5	34.2	27.6	-0.0314	50.4800	0.02698	0.08095
Eu	516	520	518	5	575	593	584.0	19	726	730	728	5	34.2	27.6	-0.0314	50.4800	0.03800	0.11401
Tb	516	520	518	5	688	708	698.0	21	726	730	728	5	34.2	27.6	-0.0314	50.4800	0.02563	0.07690
Ge MS	725	734	518	10	978	999	988.5	22	1136	1140	1138	5	25.7	10.6	-0.0244	38.3158		
<b>SP4</b>																		
Zn	767	771	769	5	854	875	864.5	22	891	895	893	5	24.2	25.4	0.0097	16.7581	0.01733	0.05200
Ga	891	895	893	5	915	936	925.5	22	1017	1021	1019	5	25.4	25.6	0.0016	23.9825	0.01483	0.04450
Ge	891	895	893	5	978	999	988.5	22	1017	1021	1019	5	25.4	25.6	0.0016	23.9825	0.01298	0.03895
As	1017	1021	1019	5	1044	1065	1054.5	22	1153	1157	1155	5	25.6	21.8	-0.0279	54.0721	0.01166	0.03497
Se	1017	1021	1019	5	1111	1133	1122.0	23	1153	1157	1155	5	25.6	21.8	-0.0279	54.0721	0.01086	0.03257
Br	1153	1157	1155	5	1181	1204	1192.5	24	1227	1231	1229	5	21.8	19.2	-0.0351	62.3811	0.00991	0.02973
Rb	1299	1303	1301	5	1327	1352	1339.5	26	1376	1379	1377.5	4	26.0	37.8	0.1536	-173.8268	0.00850	0.02550
Sr	1376	1379	1377.5	4	1404	1430	1417.0	27	1454	1458	1456	5	37.8	67.8	0.3828	-489.5605	0.00784	0.02351
Y	1454	1458	1456	5	1481	1508	1494.5	28	1533	1537	1535	5	67.8	105.0	0.4709	-617.8101	0.00733	0.02200
Zr	1533	1537	1535	5	1562	1590	1576.0	29	1615	1619	1617	5	105.0	158.2	0.6488	-890.8780	0.00678	0.02034
Hg	891	895	893	5	989	1010	999.5	22	1017	1021	1019	5	25.4	25.6	0.0016	23.9825	0.02201	0.06604
Pb	1222	1231	1226.5	10	1252	1273	1262.5	22	1296	1305	1300.5	10	20.9	29.4	0.1149	-119.9818	0.02563	0.07688
<b>SP5</b>																		
Pd	2061	2080	2070.5	20	2095	2130	2112.5	36	2145	2164	2154.5	20	13.4	14.5	0.0137	-14.9961	0.01458	0.04373
Ag	2158	2177	2167.5	20	2192	2229	2210.5	38	2244	2263	2253.5	20	13.6	13.7	0.0012	11.0797	0.01402	0.04205
Cd	2258	2277	2267.5	20	2292	2331	2311.5	40	2346	2365	2355.5	20	13.8	13.7	-0.0011	16.3767	0.01375	0.04125
In	2360	2379	2369.5	20	2394	2435	2414.5	42	2450	2469	2459.5	20	14.0	15.5	0.0161	-24.1753	0.01375	0.04125
Sn	2463	2482	2472.5	20	2497	2540	2518.5	44	2555	2574	2564.5	20	15.9	18.5	0.0288	-55.3688	0.01415	0.04246
Sb	2569	2588	2578.5	20	2603	2649	2626.0	47	2664	2683	2673.5	20	17.5	23.1	0.0589	-134.5458	0.01503	0.04508
Cs	3021	3040	3030.5	20	3055	3112	3083.5	58	3127	3146	3136.5	20	39.2	49.1	0.0934	-243.8373	0.02475	0.07424
Ba	3143	3162	3152.5	20	3177	3236	3206.5	60	3251	3270	3260.5	20	47.5	55.0	0.0699	-172.9331	0.03037	0.09110
La	3265	3284	3274.5	20	3299	3361	3330.0	63	3376	3395	3385.5	20	58.3	58.7	0.0041	44.9750	0.03836	0.11508

Figure 1. Final calibration data sheet

Analyte to be corrected	sp	Interferent	sp	Factor	Amount Subtracted	Corrected Net Counts	Corrected Uncertainty
K	1	Cd	5	0	0.00	0	0.00
Sn	5	Ag	5	0	0.00	110	0.00
K	1	Sn	4	0	0.00	0	0.00
Ca	1	Sn	4	0	0.00	0	0.00
Sb	5	Cd	5	0.13207	1.98	12	4.41
K	1	Sb	5	0	0.00	0	0.00
Ca	1	SB	5	0	0.00	0	0.00
Ca	1	K	1	0.07171	0.00	0	4.32
Ca	2	K	2	0.07171	5.37	45	3.05
Sc	2	Ca	2	0.092	4.12	0	4.59
Ba	5	I	5	0	0.00	10	0.00
Ti	2	BA	5	0.3332	0.00	345	0.00
Ti	2	Fe MS	2	0.0012	381.35	0	0.68
V	2	BA	5	0.1321	0.00	0	0.00
V	2	TI	2	0.13371	0.00	0	8.58
Cr	2	V	2	0.13568	0.00	0	6.69
Mn	3	CR	3	0.1257	18.74	65	4.15
Fe	3	MN	3	0.08087	5.27	415	2.50
Cu	3	Ge MS	3	0.0017	209.02	32	0.60
Pb	4	SE	4	0.07656	0.00	0	2.32
S	1	PB	4	0.11478	0.00	0	8.20
Cl	1	PB	4	0.02922	0.00	689	2.09
Cl	1	S	1	0.0175	0.00	689	0.86
Cl	1	Ti MS	1	0.0021	645.80	43	1.17
S	1	CL	1	0	0.00	0	0.00
Co	3	FE	3	0.06992	29.00	0	2.61
Sm	3	Cr	3	0.0762	11.36	0	2.51
Sm	3	Mn	3	0.0172	1.12	0	0.53
Eu	3	Cr	3	0.0611	9.11	0	2.02
Eu	3	Mn	3	0.8262	53.88	0	25.50
Tb	3	Fe	3	0.1	41.47	0	3.74
Tb	3	Co	3	0.9369	0.00	0	28.34
Co	4	FE	4	0.06007	10.75	0	2.18
Ga	4	PB	4	0.06488	0.00	28	2.16
As	4	PB	4	1.200648	0.00	0	0.00
Ge	4	ZN	3	0	0.00	0	0.00
Ge	4	Hg	4	0	0.00	0	0.00
Br	4	AS	4	0.04556	0.00	43	1.40
Rb	4	BR	4	0.13207	5.73	24	4.24
Y	4	PB	4	0.06959	0.00	0	1.78
Y	4	RB	4	0	0.00	0	0.00
Hf	4	Pb	4	0.0174	0.00	0	0.58
Hf	4	Cu	4	0.0883	13.99	0	3.05
Ga	4	Ta	4	0.65672	41.21	0	22.59

Figure 2. Overlap correction data.

KeVex Absorption Table

	Incom	Incom	Exit	Exit	Exit	Exit	Exit	Exit	Exit	Exit	Exit	Exit
	Rh La	Ti Ka	Na Ka	Mg Ka	Al Ka	Si Ka	P Ka	S Ka	Cl Ka	K Ka	Ca Ka	Ti Ka
csc	2.79	1.4142	1.4142	1.4142	1.4142	1.4142	1.4142	1.4142	1.4142	1.4142	1.4142	1.4142
C	116	26	1780	1050	640	400	263	175	125	61	45	26
O	275	63	4000	2440	1520	965	633	435	300	162	115	63
Na	650	163	8160	4925	3395	2100	1480	1030	715	366	276	163
Mg	850	212	580	350	4050	2660	1794	1330	930	490	367	212
Al	1035	263	850	500	330	3170	2223	1610	1130	595	450	263
Si	1265	328	1230	740	480	315	2645	1960	1375	735	556	328
P	1435	389	1640	1015	650	435	300	2180	1570	850	648	389
S	1770	453	2100	1320	795	525	355	250	1920	1030	671	453
Cl	190	512	2500	1570	960	635	435	300	210	1140	870	512
K	280	689	3425	2120	1300	855	593	425	305	162	606	689
Ca	320	780	3850	2380	1500	980	710	480	345	185	142	780
Ti	425	114	4680	2975	2000	1300	905	645	455	250	190	114
V	480	129	5050	3260	2200	1460	1020	730	520	280	215	129
Cr	550	153	5480	3510	2470	1670	1165	835	590	320	247	153
Mn	675	171	5895	3790	2700	1920	1310	935	665	360	279	171
Fe	700	193	6275	4100	2910	2040	1465	1070	760	410	317	193
Co	735	198	6640	4380	3070	2195	1565	1160	790	435	341	198
Ni	830	244	6800	4540	3140	2225	1675	1260	900	490	379	244
Cu	885	262	7550	5035	3450	2415	1780	1350	960	530	419	262
Zn	1050	292	0	5235	3645	2510	1915	1460	1130	595	480	292
Ga	1145	300	0	0	3810	2645	2025	1575	1225	700	508	300
Ge	1210	325	0	0	3995	2750	2145	1670	320	760	543	325
As	1310	340	1580	1020	0	2880	2490	1795	1420	820	595	340
Se	1380	380	1740	1110	0	3010	2620	1930	1530	925	660	380
Br	1410	480	1875	1190	840	0	2528	2060	1615	1025	703	480
Rb	1630	455	2200	1380	950	710	0	2330	1810	1190	805	455
Sr	1740	180	2400	1500	1020	760	0	2465	1910	1260	875	180
Y	1875	515	2555	1820	1080	810	648	0	2020	1310	938	515
Zr	1940	580	2755	1740	1155	855	688	0	2125	1390	995	580
Mo	0	720	3180	2005	1315	990	788	630	0	1540	1190	720
Pd	550	890	4100	2590	1675	1260	1000	800	590	0	683	890
Ag	585	925	4230	2700	1800	1350	1058	860	625	355	1365	925
Cd	620	1025	4660	2930	1880	1415	1120	890	665	385	0	1025
In	650	1065	4880	3080	1975	1480	1175	930	700	400	170	1065
Sn	720	1020	5300	3360	2280	1475	1235	990	760	435	348	1020
Sb	730	0	5510	3500	2355	1660	1310	1050	780	455	368	0
Te	770	0	5825	3650	2500	1740	1375	1100	825	480	385	0
I	805	265	0	3870	2645	1840	1453	1160	870	505	405	265
Cs	0	0	0	0	0	2010	0	0	0	0	0	0
Ba	940	310	0	0	0	2130	1680	1345	1010	585	473	310
La	980	325	0	0	0	2210	1760	1400	1055	610	495	325
Hg	0	880	0	0	0	0	0	0	0	0	1435	880
Pb	0	935	0	0	0	0	0	0	0	0	1500	935
Σ	35506	17549	131275	92680	72480	64755	51324	44600	37550	24576	23043	17549

Figure 3. Interelement energy absorption table (see section 9.7.2)